National Environmental Laboratory **Accreditation** Conference

QUALITY SYSTEMS

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5.0 QUALITY SYSTEMS

INTRODUCTION

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures, which shall be delineated in a Quality Manual and followed to ensure and document the quality of the analytical data. Laboratories seeking accreditation under NELAP must assure implementation of all QA policies and the essential applicable QC procedures specified in this Chapter. The QA policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity.

The intent of this Chapter is to provide sufficient detail concerning quality management requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

NELAC is committed to the use of Performance-based Measurement Systems (PBMS) in environmental testing and provides the foundation for PBMS implementation in these standards. While this standard may not currently satisfy all the anticipated needs of PBMS, NELAC will address future needs within the context of State statutory and regulatory requirements and the finalized EPA implementation plans for PBMS.

Chapter Five is organized according to the structure of ISO/IEC Guide 25, 1990. Where deemed necessary, specific areas within this Chapter may contain more information than specified by ISO/IEC Guide 25.

All items identified in this Chapter shall be available for on-site inspection or data audit.

5.1 SCOPE

a) This Standard sets out the general requirements in accordance with which a laboratory has to demonstrate that it operates, if it is to be recognized as competent to carry out specific environmental tests.

b) This Standard includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority granting the recognition (or approval).

   If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. (See the supplemental accreditation requirements in Section 1.9.2.)

c) This Standard is for use by environmental testing laboratories in the development and implementation of their quality systems. It shall be used by accrediting authorities, in assessing the competence of environmental laboratories.

5.2 REFERENCES

See Appendix A.

5.3 DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ISO 8402, ANSI/ASQC E-4,1994, the EPA "Glossary of Quality Assurance Terms and Acronyms", and the *International vocabulary of basic and general terms in metrology (VIM*) are applicable, the most relevant being quoted in Appendix A, Glossary, of Chapter One together with further definitions applicable for the purposes of this Standard.

See Appendix A, Glossary, of Chapter One.

5.4 ORGANIZATION AND MANAGEMENT

5.4.1 Legal Definition of Laboratory

The laboratory shall be legally identifiable. It shall be organized and shall operate in such a way that its permanent, temporary and mobile facilities meet the requirements of this Standard**.**

5.4.2 Organization

The laboratory shall:

a) have managerial staff with the authority and resources needed to discharge their duties;

b) have processes to ensure that its personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work;

c) be organized in such a way that confidence in its independence of judgment and integrity is maintained at all times;

d) specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests;

   Such documentation shall include:

   1) a clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured and

   2) job descriptions for all positions.

e) provide supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test and the assessment of the results;

   The ratio of supervisory to non-supervisory personnel shall be such as to ensure adequate supervision to ensure adherence to laboratory procedures and accepted techniques.

f) have a technical director(s) (however named) who has overall responsibility for the technical operation of the environmental testing laboratory;

   The technical director(s) shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.

The technical director(s) shall meet the requirements specified in the Accreditation Process. (see 4.1.1.1)

g) have a quality assurance officer (however named) who has responsibility for the quality system and its implementation;

The quality assurance officer shall have direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and to the technical director. Where staffing is limited, the quality assurance officer may also be the technical director or deputy technical director;

The quality assurance officer (and/or his/her designees) shall:

1) serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

2) have functions independent from laboratory operations for which they have quality assurance oversight;

3) be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

4) have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC;

5) have a general knowledge of the analytical test methods for which data review is performed;

6) arrange for or conduct internal audits on the entire technical operation annually; and,

7) notify laboratory management of deficiencies in the quality system and monitor corrective action.

h) nominate deputies in case of absence of the technical director(s) and/or quality assurance officer;

i) have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights (this may not apply to in-house laboratories);

j) when available, participate in interlaboratory comparisons and proficiency testing programs. For purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in Chapter Two.

5.5 QUALITY SYSTEM - ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS AND DATA VERIFICATION

5.5.1 Establishment

The laboratory shall establish and maintain a quality system based on the required elements contained in this chapter and appropriate to the type, range and volume of environmental testing activities it undertakes.

a) The elements of this quality system shall be documented in the organization's quality manual.

b) The quality documentation shall be available for use by the laboratory personnel.

c) The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

d) The laboratory management shall ensure that these policies and objectives are documented in a quality manual and communicated to, understood, and implemented by all laboratory personnel concerned.

e) The quality manual shall be maintained current under the responsibility of the quality assurance officer.

5.5.2 Quality Manual

The quality manual, and related quality documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this Standard.

The Quality Manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different from above), and telephone number of individual(s) responsible for the laboratory; the name of the quality assurance officer (however named); the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;

The quality manual and related quality documentation shall also contain:

a) a quality policy statement, including objectives and commitments, by top management;

b) the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;

c) the relationship between management, technical operations, support services and the quality system;

d) procedures to ensure that all records required under this Chapter are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;

e) job descriptions of key staff and reference to the job descriptions of other staff;

f) identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed and dated concurrence, (with appropriate titles) of all responsible parties including the QA officer(s), technical director(s), and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager;

g) the laboratory's procedures for achieving traceability of measurements;

h) a list of all test methods under which the laboratory performs its accredited testing;

i) mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

j) reference to the calibration and/or verification test procedures used;

k) procedures for handling submitted samples;

l) reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;

m) reference to procedures for calibration, verification and maintenance of equipment;

n) reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

o) procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

p) the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

q) procedures for dealing with complaints;

r) procedures for protecting confidentiality (including national security concerns), and proprietary rights;

s) procedures for audits and data review;

t) processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;

u) processes/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions;

v) reference to procedures for reporting analytical results; and,

w) a Table of Contents, and applicable lists of references and glossaries, and appendices.

5.5.3 Audits

5.5.3.1 Internal Audits

The laboratory shall arrange for annual internal audits to verify that its operations continue to comply with the requirements of the laboratory's quality system. It is the responsibility of the quality assurance officer to plan and organize audits as required by a predetermined schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out. Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work may have been affected.

5.5.3.2 Managerial Review

The laboratory management shall conduct a review, at least annually, of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review shall take account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of interlaboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and other relevant factors. The laboratory shall have a procedure for review by management and maintain records of review findings and actions.

5.5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them shall be documented. The laboratory management shall ensure that these actions are discharged within the agreed time frame.

5.5.3.4 Performance Audits

In addition to periodic audits, the laboratory shall ensure the quality of results provided to clients by implementing checks to monitor the quality of the laboratory's analytical activities. Examples of such checks are:

a) internal quality control procedures using whenever possible statistical techniques; (see 5.5.4 below)

b) participation in proficiency testing or other interlaboratory comparisons (See Chapter Two);

c) use of certified reference materials and/or in-house quality control using secondary reference materials as specified in Section 5.5.4;

d) replicate testings using the same or different test methods;

e) re-testing of retained samples;

f) correlation of results for different parameters of a sample (for example, total phosphorus should be greater than or equal to orthophosphate).

5.5.3.5 Corrective Actions

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method Standard Operating Procedures (see 5.10.1.1), the laboratory shall implement general procedures to be followed to determine when departures from documented policies, procedures and quality control have occurred. These procedures shall include but are not limited to the following:

1) identify the individual(s) responsible for assessing each QC data type;

2) identify the individual(s) responsible for initiating and/or recommending corrective actions;

3) define how the analyst should treat a data set if the associated QC measurements are unacceptable;

4) specify how out-of-control situations and subsequent corrective actions are to be documented; and,

5) specify procedures for management (including the QA officer) to review corrective action reports.

b) To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s).

5.5.4 Essential Quality Control Procedures

These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, whole effluent toxicity, microbiological, radiological, air) and are further described in Appendix D. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have protocols in place to monitor the following quality controls:

1) Adequate positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

2) Adequate tests to define the variability and/or repeatability of the laboratory results such as replicates;

3) Measures to assure the accuracy of the test method including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

4) Measures to evaluate test method capability, such as detection limits and quantitation limits or range of applicability such as linearity;

5) Selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;

6) Selection and use of reagents and standards of appropriate quality;

7) Measures to assure the selectivity of the test for its intended purpose; and

8) Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method such as temperature, humidity, light, or specific instrument conditions.

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria shall be used to determine the usability of the data. (See Appendix D.)

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See 5.11.2, Sample Acceptance Policy.)

d) The quality control protocols specified by the laboratory's method manual (5.10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals.

The essential quality control measures for testing are found in Appendix D of this Chapter.

5.6 PERSONNEL

5.6.1 General Requirements for Laboratory Staff

The laboratory shall have sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned functions.

All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures and records management.

5.6.2 Laboratory Management Responsibilities

In addition to 5.4.2.d, the laboratory management shall be responsible for:

a) Defining the minimal level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance, colony counting, aseptic or quantitative techniques shall be considered;

b) Ensuring that all technical laboratory staff have demonstrated capability in the activities for which they are responsible. Such demonstration shall be documented. (See Appendix C);

Note: In laboratories with specialized “work cells” (a well defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

c) Ensuring that the training of each member of the technical staff is kept up-to-date (on-going) by the following:

1) Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

2) Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented.

3) Training courses in ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions. Evidence must also be on file which demonstrates that each employee has read, acknowledged and understood their personal ethical and legal responsibilities including the potential punishments and penalities for improper, unethical or illegal actions.

4) Analyst training shall be considered up to date if an employee training file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure) and documentation of continued proficiency by at least one of the following once per year:

i. Acceptable performance of a blind sample (single blind to the analyst);

ii. Another demonstration of capability;

iii. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5035/8260) would only require documentation for one of the test methods;

iv. At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;

v. If i-iv cannot be performed, analysis of authentic samples that have been analyzed by another trained analyst with statistically indistinguishable results.

d) Documenting all analytical and operational activities of the laboratory;

e) Supervising all personnel employed by the laboratory;

f) Ensuring that all sample acceptance criteria (Section 5.11) are verified and that samples are logged into the sample tracking system and properly labeled and stored;

g) Documenting the quality of all data reported by the laboratory; and

h) Developing a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program could include: internal proficiency testing (single and double blind); post-analysis, electronic data and magnetic tape audits; effective reward program to improve employee vigilance and co-monitoring; and separate SOPs identifying appropriate and inappropriate laboratory and instrument manipulation practices.

5.6.3 Records

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory [see 5.6.2.c], including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in 5.10.2.1 for chemical testing.

5.7 PHYSICAL FACILITIES - ACCOMMODATION AND ENVIRONMENT

5.7.1 Environment

a) Laboratory accommodation, test areas, energy sources, lighting, heating and ventilation shall be such as to facilitate proper performance of tests.

b) The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement. Particular care shall be taken when such activities are undertaken at sites other than the permanent laboratory premises.

c) The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate. Such environmental conditions may include biological sterility, dust, electromagnetic interference, humidity, mains voltage, temperature, and sound and vibration levels .

d) In instances where monitoring or control of any of the above mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

NOTE: It is the laboratory's responsibility to comply with the relevant health and safety requirements. This aspect, however, is outside the scope of this Standard.

5.7.2 Work Areas

a) There shall be effective separation between neighboring areas when the activities therein are incompatible including culture handling or incubation areas and volatile organic chemicals handling areas.

b) Access to and use of all areas affecting the quality of these activities shall be defined and controlled.

c) Adequate measures shall be taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

d) Work spaces must be available to ensure an unencumbered work area. Work areas include:

1) access and entryways to the laboratory;
2) sample receipt area(s);
3) sample storage area(s);
4) chemical and waste storage area(s); and,
5) data handling and storage area(s).

5.8 EQUIPMENT AND REFERENCE MATERIALS

a) The laboratory shall be furnished with all items of equipment (including reference materials) required for the correct performance of tests for which accreditation is sought. In those cases where the laboratory needs to use equipment outside its permanent control it shall ensure that the relevant requirements of this Standard are met.

b) All equipment shall be properly maintained, inspected and cleaned. Maintenance procedures shall be documented.

c) Any item of the equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or otherwise to be defective, shall be taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

d) Each item of equipment including reference materials shall, when appropriate, be labeled, marked or otherwise identified to indicate its calibration status.

e) Records shall be maintained of each major item of equipment and all reference materials significant to the tests performed. These records shall include documentation on all routine and non-routine maintenance activities and reference material verifications.

The records shall include:

1) the name of the item of equipment;
2) the manufacturer's name, type identification, and serial number or other unique identification;
3) date received and date placed in service (if available);
4) current location, where appropriate;
5) if available, condition when received (e.g. new, used, reconditioned);
6) copy of the manufacturer's instructions, where available;
7) dates and results of calibrations and/or verifications and date of the next calibration and/or verification;
8) details of maintenance carried out to date and planned for the future; and,
9) history of any damage, malfunction, modification or repair.

5.9 MEASUREMENT TRACEABILITY AND CALIBRATION

5.9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests shall be calibrated and/or verified before being put into service and on a continuing basis. The laboratory shall have an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers and control standards.

5.9.2 Traceability of Calibration

a) The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that, wherever applicable, measurements made by the laboratory are traceable to national standards of measurement where available.

b) Calibration certificates, when available, shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.

c) Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.

5.9.3 Reference Standards

a) Reference standards of measurement held by the laboratory (such as Class S or equivalent weights or traceable thermometers) shall be used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards have not been invalidated. Reference standards of measurement shall be calibrated by a body that can provide, where possible, traceability to a national standard of measurement.

b) There shall be a program of calibration and verification for reference standards.

c) Where relevant, reference standards and measuring and testing equipment shall be subjected to in-service checks between calibrations and verifications. Reference materials shall, where possible, be traceable to national or international standards of measurement, or to national or international standard reference materials.

5.9.4 Calibration

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and 2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

5.9.4.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All support equipment shall be:

a) Maintained in proper working order. The records of all repair and maintenance activities including service calls, shall be kept.

b) Calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which this equipment is used or:

   1) The equipment shall be removed from service until repaired; or

   2) The laboratory shall maintain records of established correction factors to correct all measurements.

c) Raw data records shall be retained to document equipment performance.

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, incubators and water baths shall be checked with NIST traceable references (where possible) in the expected use range. Additional monitoring as prescribed by the test method shall be performed for any device that is used in a critical test (such as incubators or water baths). The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

e) Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.

f) For chemical tests the temperature, cycle time, and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

g) For biological tests the sterilization temperature, cycle time, sterilization time, and pressure of each run of autoclaves must be documented by the use of appropriate chemical or biological sterilization indicators. Autoclave tape may be used to indicate by color change that a load has been processed, but not to demonstrate completion of an acceptable sterilization cycle. Demonstration of sterilization shall be provided by a continuous temperature recording or with the frequent use of spore strips.

5.9.4.2 Instrument Calibration:

This standard specifies the essential elements that will define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data will be of known quality and be appropriate for a given regulation or decision. This standard does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are to be followed.

Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.

5.9.4.2.1 Initial Instrument Calibration:

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, and acceptance criteria associated statistics must be included or referenced in the test method SOP.

b) Sufficient raw data records must be retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor.

c) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

d) All initial instrument calibrations must be verified with a standard obtained from a second source and traceable to a national standard, when available.

e) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient or relative percent difference.

f) Results of samples not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags or explained in the case narrative. The lowest calibration standard must be above the detection limit.

g) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed. Data associated with an unacceptable initial instrument calibration shall not be reported.

h) Calibration standards must include concentrations at or below the regulatory limit/decision level, if these limits/levels are known by the laboratory, unless these concentrations are below the laboratory's demonstrated detection limits (See D.1.4 Detection Limits)

i) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, not including blanks or a zero standard. The laboratory must have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

5.9.4.2.2 Continuing Instrument Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. The concentrations of the calibration verification shall be varied within the established calibration range. If an internal standard is used, only one continuing instrument calibration verification must be analyzed per analytical batch.

c) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor.

d) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.

e) If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. If the laboratory has not demonstrated acceptable performance, sample analyses shall not occur until a new initial calibration curve is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

   i. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

   ii. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed

a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

5.10 TEST METHODS AND STANDARD OPERATING PROCEDURES

5.10.1 Methods Documentation

a) The laboratory shall have documented instructions on the use and operation of all relevant equipment, on the handling and preparation of samples and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.

b) All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available to the staff.

5.10.1.1 Standard Operating Procedures (SOPs)

Laboratories shall maintain standard operating procedures that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents.

b) The test methods may be copies of published methods as long as any changes in the methods are documented and included in the methods manual (see 5.10.1.2).

c) Copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be organized .

e) Each SOP shall clearly indicate the effective date of the document, the revision number and the signature(s) of the approving authority.

5.10.1.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.

b) This manual may consist of copies of published or referenced test methods or standard operating procedures that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each test method shall include or reference where applicable:

1) identification of the test method;
2) applicable matrix or matrices;
3) detection limit;
4) scope and application, including components to be analyzed;
5) summary of the test method;

6) definitions;
7) interferences;
8) safety;
9) equipment and supplies;
10) reagents and standards;
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measures;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and,
23) any tables, diagrams, flowcharts and validation data.

5.10.2 Test Methods

a) The laboratory shall use appropriate test methods and procedures for all tests and related activities within its responsibility (including sample collection, sample handling, transport and storage, sample preparation and sample analysis). The method and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.

1) When the use of specific test methods for a sample analysis are mandated or requested, only those methods shall be used.

2) Where test methods are employed that are not required, as in the Performance Based Measurement System approach, the methods shall be fully documented and validated (see 5.10.2.1 and Appendix C), and be available to the client and other recipients of the relevant reports.

5.10.2.1 Demonstration of Capability

a) Prior to acceptance and institution of any test method, satisfactory demonstration of method capability is required. (See Appendix C and 5.6.2.b.) In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids, biological tissue and air. In addition, for analytes which do not lend themselves to spiking, the demonstration of capability may be performed using quality control samples.

b) Thereafter, continuing demonstration of method performance, as per the quality control requirements in Appendix D (such as laboratory control samples) is required.

c) In all cases, the appropriate forms such as the Certification Statement (Appendix C) must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the Certification Statement must be retained by the laboratory. (See Appendix C for Certification Statement.)

d) A demonstration of capability must be completed each time there is a significant change in instrument type, personnel, or test method.

e) In laboratories with a specialized "work cell(s)" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability must be fully documented.

f) When a work cell(s) is employed, and the members of the cell change, the new employee(s) must work with experienced analyst(s) in the speciality area and this new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance must be documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed/replaced, the work cell must repeat the demonstration of capability (Appendix C).

g) When a work cell(s) is employed the performance of the group must be linked to the training record of the individual members of the work cell (see section 5.6.2).

5.10.3 Sample Aliquots

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples.

5.10.4 Data Verification

Calculations and data transfers shall be subject to appropriate checks.

a) The laboratory shall establish Standard Operating Procedure to ensure that the reported data are free from transcription and calculation errors.

b) The laboratory shall establish a Standard Operating Procedures to ensure that all quality control measures are reviewed, and evaluated before data are reported.

5.10.5 Documentation and Labeling of Standards and Reagents

Documented procedures shall exist for the purchase, reception and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records for all standards including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt,

recommended storage conditions, and an expiration date after which the material shall not be used unless it is verified by the laboratory.

b) Original containers (such as provided by the manufacturer or vendor) shall be labeled with an expiration date.

c) Records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

d) All containers of prepared reagents and standards must bear a unique identifier and expiration date and be linked to the documentation requirements in 5.10.5.c above.

5.10.6 Computers and Electronic Data Related Requirements

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data, the laboratory shall ensure that:

a) all requirements of this Standard (i.e. Chapter Five) are met;

Sections 8.1 through 8.11 of the EPA Document “2185 - Good Automated Laboratory Practices” (1995), shall be adopted as the standard for all laboratories employing microprocessors, computers, as well as laboratories employing Laboratory Information Management Systems.

b) computer software is documented and adequate for use;

c) procedures are established and implemented for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission and data processing;

d) computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data; and,

e) it establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

5.11 SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY AND SAMPLE RECEIPT

While the laboratory may not have control of field sampling activities, the following are essential to ensure the validity of the laboratory’s data.

5.11.1 Sample Tracking

a) The laboratory shall have a documented system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of such items at any time. This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to

each sample container received in the laboratory. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

b) This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

c) The laboratory ID code shall be placed on the sample container as a durable label.

d) The laboratory ID code shall be entered into the laboratory records (see 5.11.3.d) and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration.

e) In cases where the sample collector and analyst are the same individual or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

5.11.2 Sample Acceptance Policy

The laboratory shall have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted. Data from any samples which do not meet the following criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collection personnel and shall include, but is not limited to, the following areas of concern:

a) Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) Proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) Use of appropriate sample containers;

d) Adherence to specified holding times;

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and

f) Procedures to be used when samples which show signs of damage or contamination.

5.11.3 Sample Receipt Protocols

a) Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition as prescribed in the relevant test method, shall be recorded. All items specified in 5.11.2 above shall be checked.

1) All samples which require thermal preservation shall be considered acceptable if the arrival temperature is either within +/-2°C of the required temperature or the

method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis.

b) The results of all checks shall be recorded.

c) Where there is any doubt as to the item's suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, the laboratory should consult the client for further instruction before proceeding. The laboratory shall establish whether the sample has received all necessary preparation, or whether the client requires preparation to be undertaken or arranged by the laboratory. If the sample does not meet the sample receipt acceptance criteria listed in 5.11.3.a, 5.11.3.b or 5.11.3.c, the laboratory shall either:

a) Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

2) Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

i. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

ii. The analysis data shall be appropriately "qualified" on the final report.

d) The laboratory shall utilize a permanent chronological record such as a log book or electronic database to document receipt of all sample containers.

1) This sample receipt log shall record the following:

i. Client/Project Name,

ii. Date and time of laboratory receipt,

iii. Unique laboratory ID code (see 5.11.1), and,

iv. Signature or initials of the person making the entries.

2) During the log-in process, the following information must be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request and readily available to

individuals who will process the sample. Note: the placement of the laboratory ID number on the sample container is not considered a permanent record.

i. The field ID code which identifies each container must be linked to the laboratory ID code in the sample receipt log.

ii. The date and time of sample collection must be linked to the sample container and to the date and time of receipt in the laboratory.

iii. The requested analyses (including applicable approved test method numbers) must be linked to the laboratory ID code.

iv. Any comments resulting from inspection for sample rejection shall be linked to the laboratory ID code.

e) All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

f) A complete chain of custody record (Section 5.12.4), if utilized, shall be maintained.

5.11.4 Storage Conditions

The laboratory shall have documented procedures and appropriate facilities to avoid deterioration, contamination, or damage to the sample during storage, handling, preparation, and testing; any relevant instructions provided with the item shall be followed. Where items have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored and recorded where necessary.

a) Samples shall be stored according to the conditions specified by preservation protocols:

1) Samples which require thermal preservation shall be stored under refrigeration which is +/-2° of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.

2) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.

b) Sample fractions, extracts, leachates and other sample preparation products shall be stored according to 5.11.4.a above or according to specifications in the test method.

c) Where a sample or portion of the sample is to be held secure (for example, for reasons of record, safety or value, or to enable check calibrations or tests to be performed later), the laboratory shall have storage and security arrangements that protect the condition and integrity of the secured items or portions concerned.

5.11.5 Sample Disposal

The laboratory shall have standard operating procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products.

5.12 RECORDS

The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records which document all laboratory activities. The laboratory shall retain on record all original observations, calculations and derived data, calibration records and a copy of the test report for a minimum of five years.

There are two levels of record keeping: 1) sample custody or tracking and 2) legal or evidentiary chain of custody. All essential requirements for sample custody are outlined in Sections 5.12.1, 5.12.2 and 5.12.3. The basic requirements for legal chain of custody (if required or implemented) are specified in Section 5.12.4.

5.12.1 Record Keeping System and Design

The record keeping system must allow historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

a) The records shall include the identity of personnel involved in sampling, preparation, calibration or testing.

b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.

c) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

d) All documentation entries shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records such as “sampled by”, “prepared by”, or “reviewed by”).

e) All generated data except those that are generated by automated data collection systems, shall be recorded directly, promptly and legibly in permanent ink.

f) Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

g) Refer to 5.10.6 for Computer and Electronic Data.

5.12.2 Records Management and Storage

a) All records (including those pertaining to calibration and test equipment), certificates and reports shall be safely stored, held secure and in confidence to the client. NELAP-related records shall be available to the accrediting authority.

b) All records, including those specified in 5.12.3 and 5.12.4, shall be retained for a minimum of five years from last use. All information necessary for the historical reconstruction of data must be maintained by the laboratory. Records which are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

c) Records that are stored or generated by computers or personal computers shall have hard copy or write-protected backup copies.

d) The laboratory shall establish a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation storage and reporting.

e) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

f) The laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients' instructions (see 4.1.8.e) in the event that a laboratory transfers ownership or goes out of business.

5.12.3 Laboratory Sample Tracking

5.12.3.1 Sample Handling

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to:

a) Sample preservation including appropriateness of sample container and compliance with holding time requirement;

b) Sample identification, receipt, acceptance or rejection and log-in;

c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records;

d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

e) Sample analysis;

f) Standard and reagent origin, receipt, preparation, and use;

g) Equipment receipt, use, specification, operating conditions and preventative maintenance;

h) Calibration criteria, frequency and acceptance criteria;

i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

j) Method performance criteria including expected quality control requirements;

k) Quality control protocols and assessment;

l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

m) All automated sample handling systems; and,

n) The laboratory shall have documented procedures for the receipt, retention or safe disposal of calibration or test items, including all provisions necessary to protect the integrity of the laboratory.

5.12.3.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following shall be retained:

a) All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

b) A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

c) Copies of final reports;

d) Archived standard operating procedures;

e) Correspondence relating to laboratory activities for a specific project;

f) All corrective action reports, audits and audit responses;

g) Proficiency test results and raw data; and,

h) Data review and cross checking.

5.12.3.3 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

a) Laboratory sample ID code;

b) Date and time of analysis;

c) Instrumentation identification and instrument operating conditions/parameters (or reference to such data);

d) Analysis type;

e) All manual calculations; and,

f) Analyst's or operator's initials/signature.

5.12.3.4 Administrative Records

The following shall be maintained:

a) Personnel qualifications, experience and training records;

b) Records of demonstration of capability for each analyst; and

c) A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

5.12.4 Legal/Evidentiary Custody

The use of legal chain of custody (COC) protocols may be required by some State or federal programs. In addition to the records listed in 5.12.3 and the performance standards outlined in 5.12.1 and 5.12.2, the following protocols shall be incorporated if legal COC is implemented by the organization.

5.12.4.1 Basic Requirements

The legal chain of custody records shall establish an intact, continuous record of the physical possession, storage and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For ease of discussion, the above-mentioned items shall be referred to as samples:

a) A sample is in someone's custody if:

   1) It is in one's actual physical possession;

   2) It is in one's view, after being in one's physical possession;

   3) It is in one's physical possession and then locked up so that no one can tamper with it;

   4) It is kept in a secured area, restricted to authorized personnel only.

b) The COC records shall account for all time periods associated with the samples.

c) The COC records shall identify individuals who physically handled individual samples.

d) In order to simplify record-keeping, the number of people who physically handle the sample should be minimized. A designated sample custodian, who is responsible for receiving, storing and distributing samples is recommended.

e) The COC records are not limited to a single form or document. However, organizations should attempt to limit the number of documents that would be required to establish COC.

f) Legal chain of custody shall begin at the point established by the federal or State oversight program. This may begin at the point that cleaned sample containers are provided by the laboratory or the time sample collection occurs.

g) The COC forms shall remain with the samples during transport or shipment.

h) If shipping containers and/or individual sample containers are submitted with sample custody seals, and any seals are not intact, the lab shall note this on the chain of custody.

i) Mailed packages should be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent chain-of-custody documentation.

j) Once received by the laboratory, laboratory personnel are responsible for the care and custody of the sample and must be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses are completed or the time of sample disposal.

5.12.4.2 Required Information in Custody Records

In addition to the information specified in 5.11.1.a and 5.11.1.b, tracking records shall include, by direct entry or linkage to other records:

a) Time of day and calendar date of each transfer or handling procedure;

b) Signatures of all personnel who physically handle the sample(s);

c) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis and reporting; and

d) Common carrier documents.

5.12.4.3 Controlled Access to Samples

Access to all legal samples and subsamples shall be controlled and documented.

a) A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a custody room.

b) Where possible, distribution of samples to the analyst performing the analysis must be made by the custodian(s).

c) The laboratory area must be maintained as a secured area, restricted to authorized personnel only.

d) Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample must be retained in the custody room until permission to destroy the sample is received by the custodian or other authority.

5.12.4.4 Transfer of Samples to Another Party

Transfer of samples, subsamples, digestates or extracts to another party are subject to all of the requirements for legal chain of custody.

5.12.4.5 Sample Disposal

a) If the sample is part of litigation, disposal of the physical sample shall occur only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample.

b) All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.

c) Records shall indicate the date of disposal, the nature of disposal (such as sample depleted, sample disposed in hazardous waste facility, or sample returned to client), and the name of the individual who performed the task.

5.13 LABORATORY REPORT FORMAT AND CONTENTS

The results of each test, or series of tests carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively. The results shall normally be reported in a test report and shall include all the information necessary for the interpretation of the test results and all information required by the method used. Some regulatory reporting requirements or formats such as monthly operating reports, may not require all items listed below, however, the laboratory shall provide all the required information to their client for use in preparing such regulatory reports.

a) Except as discussed in 5.13.b, each report to an outside client shall include at least the following information (those prefaced with “where relevant” are not mandatory):

1) a title, e.g., "Test Report", or "Test Certificate", "Certificate of Results" or "Laboratory Results";

2) name and address of laboratory, and location where the test was carried out if different from the address of the laboratory and phone number with name of contact person for questions;

3) unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;

This requirement may be presented in several ways:

i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

ii. Each page is identified with the unique report identification, the pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

4) name and address of client, where appropriate and project name if applicable;

5) description and unambiguous identification of the tested sample including the client identification code;

6) identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

7) date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 48 hours;

8) identification of the test method used, or unambiguous description of any non-standard method used;

9) if the laboratory collected the sample, reference to sampling procedure;

10) any deviations from (such as failed quality control), additions to or exclusions from the test method (such as environmental conditions), and any non-standard conditions that may have affected the quality of results, and including the use and definitions of data qualifiers;

11) measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units such as μg/l or mg/kg; and for Whole Effluent Toxicity, identify the statistical package used to provide data;

12) when required, a statement of the estimated uncertainty of the test result;

13) a signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;

14) at the laboratory's discretion, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;

15) at the laboratory's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;

16) clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc; and,

17) clear identification of numerical results with values outside of quantitation levels.

b) Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in 1 through 17 above readily available for review by the accrediting authority. However formal reports detailing the information are not required if:

1) The in-house laboratory is itself responsible for preparing the regulatory reports; or

2) The laboratory provides information to another individual within the organization for preparation of regulatory reports. The facility management must ensure that the appropriate report items are in the report to the regulatory authority if such information is required.

c) Where the certificate or report contains results of tests performed by subcontractors, these results shall be clearly identified by subcontractor name or applicable accreditation number.

d) After issuance of the report, the laboratory report shall remain unchanged. Material amendments to a calibration certificate, test report or test certificate after issue shall be made only in the form of a further document, or data transfer including the statement "Supplement to Test Report or Test Certificate, serial number . . . [or as otherwise identified]", or equivalent form of wording. Such amendments shall meet all the relevant requirements of this Standard.

e) The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate or amendment to a report or certificate.

f) The laboratory shall ensure that, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will follow documented procedures that ensure that the requirements of this Standard are met and that confidentiality is preserved.

g) Laboratories accredited to be in compliance with these standards shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

5.14 SUBCONTRACTING ANALYTICAL SAMPLES

a) The laboratory shall advise the client in writing of its intention to subcontract any portion of the testing to another party.

b) Where a laboratory subcontracts any part of the testing covered under NELAP, this work shall be placed with a laboratory accredited under NELAP for the tests to be performed.

c) The laboratory shall retain records demonstrating that the above requirements have been met.

5.15 OUTSIDE SUPPORT SERVICES AND SUPPLIES

a) Where the laboratory procures outside services and supplies, other than those referred to in this Standard, in support of tests, the laboratory shall use only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests.

b) Where no independent assurance of the quality of outside support services or supplies is available, the laboratory shall have procedures to ensure that purchased equipment, materials and services comply with specified requirements. The laboratory should, wherever possible, ensure that purchased equipment and consumable materials are not used until they have been inspected, calibrated or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned.

c) The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for tests.

5.16 COMPLAINTS

The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this Standard or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with Section 5.5.3.1. Records of the complaint and subsequent actions shall be maintained.

QUALITY SYSTEMS
APPENDIX A

REFERENCES

Appendix A - REFERENCES

40 CFR Part 136, Appendix A, paragraphs 8.1.1 and 8.2

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ISO Guide 9000: 1994 Quality management and quality assurance standards - Guidelines for selection and use

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Manual of Method for General Bacteriology, Philipp Gerhard et al., American Society for Microbiology, Washington, 1981

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APPENDIX B

(Reserved)

QUALITY SYSTEMS
APPENDIX C

DEMONSTRATION OF CAPABILITY

Appendix C - DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a significant change in instrument type, personnel or test method (see 5.10.2.1).

Note: In laboratories with specialized "work cells" (a well defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids, biological tissue and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e.,at least four consecutive matrix spikes within the last twelve months. In addition, for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the form in this appendix.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, shall be performed if required by mandatory test method or regulation. Note: For analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration. It is the responsibility of the laboratory to document that other approaches to DOC are adequate, this shall be documented in the laboratory's Quality Manual.

a) A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery ($\bar{x}$) in the appropriate reporting units (such as μg/L) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence absence and logarithmic values, the laboratory will assess performance against established and documented criteria.

e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the

parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee (see 5.6.3 and 5.12.3.4.b).

Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix:
(examples: laboratory pure water, soil, air, solid, biological tissue)
Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____	_____	_____
Technical Director's Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

QUALITY SYSTEMS
APPENDIX D

ESSENTIAL QUALITY CONTROL REQUIREMENTS

Appendix D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

The quality control protocols specified by the laboratory's method manual (5.10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals.

All quality control measures shall be assessed and evaluated on an on-going basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter Five, e.g., 5.5.4, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, W.E.T. testing, microbiology testing, radiochemical testing and air testing.

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

a) Negative Controls

1) Method Blanks - Shall be performed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if

i) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or

ii) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

b) Positive Controls

1) Laboratory Control Sample (LCS) - (QC Check Samples) Shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance. NOTE: the matrix spike (see 2 below) may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS.

2) Matrix Spikes (MS) - Shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total

dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

4) If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

D.1.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates - Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Analytical Capability - (Section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

b) Calibration - Calibration protocols specified in Section 5.9.4 shall be followed.

c) Proficiency Test Samples - The results of such analyses (5.4.2.j or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.1.4 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the

mandated test method or applicable regulation, e.g., MDL. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

a) A detection limit study is not required for any component for which spiking solutions or quality control samples are not available such temperature.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

c) Detection limits must be determined each time there is a significant change in the test method or instrument type.

d) It is essential that all sample processing steps of the analytical method be included in the determination of the detection limit.

e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

f) The laboratory must have established procedures to tie detection limits with quantitation limits.

D.1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.6 Quality of Standards and Reagents

a) The source of standards shall comply with 5.9.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

D.1.7 Selectivity

a) Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.

b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

c) The laboratory shall document acceptance criteria for mass spectral tuning.

D.1.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.2 WHOLE EFFLUENT TOXICITY TESTING

D.2.1 Positive and Negative Controls

a) Positive Control - Reference Toxicants - Reference toxicant tests indicate the sensitivity of the test organisms being used and demonstrate a laboratory's ability to obtain consistent results with the test method.

1) The laboratory must demonstrate its ability to obtain consistent results with reference toxicants before it performs toxicity tests with effluents for permit compliance purposes.

i. An intralaboratory coefficient of variation (%CV) is not established for each test method. However, a testing laboratory shall maintain control charts for the control performance and reference toxicant statistical endpoint (such as NOEC or ECp) and shall evaluate the intralaboratory variability with a specific reference toxicant for each test method. In addition, a laboratory must produce test results that meet test acceptability criteria (such as greater than 80% survival in the control) as specified in the specific test method.

ii. Intralaboratory precision on an ongoing basis must be determined through the use of reference toxicant tests and plotted in quality control charts. As specified in the test methods, the control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC 50 for acute tests, over time within a laboratory.

2) The frequency of reference toxicant testing shall comply with the EPA or State permitting authority requirements.

3) The USEPA test methods for EPA/600/4-91-002, EPA/600/4-91-003 and EPA/600/4-90-027F do not currently specify a particular reference toxicant and dilution series, however, if the State or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements.

4) Test Acceptability Criteria (TAC) - The test acceptability criteria (for example, the chronic *Ceriodaphnia* test, requires 80% or greater survival and an average 15 young per female in the controls) as specified in the test method must be achieved for both the reference toxicant and effluent test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity:

   i. The control population of *Ceriodaphnia* shall contain no more than 20% males.

   ii. An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical employee and the permitting authority.

b) Negative Control - Control, Brine Control or Dilution Water - The standards for the use, type and frequency of testing are specified by the test methods and by permit and shall be followed.

D.2.2 Variability and/or Reproducibility

Intralaboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item D.2.1.a above.

D.2.3 Accuracy

This principle is not applicable to Whole Effluent Toxicity.

D.2.4 Test Sensitivity

a) Test sensitivity (or test power) of the tests will depend in part on the number of replicates per concentration, the significance level selected (0.05), and the type of statistical analysis. If the variability remains constant, the sensitivity of the test will increase as the number of replicates is increased. Test sensitivity is the minimum significant difference (MSD) between the control and test concentration that is statistically significant. If the Dunnett's procedure is used, the MSD shall be calculated according to the formula specified by the EPA test method and reported with the test results.

b) Estimate the MSD for non-normal distribution and or heterogenous variances.

c) Point estimates: (LCp, ICp, or ECp) - Confidence intervals shall be reported as a measure of the precision around the point estimate value.

d) The MSD shall be calculated and reported for only chronic endpoints. In addition, the calculated endpoint is typically a lethal concentration of 50% (LC 50), therefore, confidence intervals shall be reported as a measure of the precision around the point estimate value. In order to have sufficient replicates to perform a reliable MSD, such tests shall have a minimum of four replicates per treatment so that either parametric or non parametric tests can be conducted.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) The methods of data analysis and endpoints will be specified by language in the permit or, if not present in the permit, by the EPA methods manuals for Whole Effluent Toxicity.

b) Dose Response Curves - When required, the data shall be plotted in the form of a curve relating the dose of the chemical to cumulative percentage of test organisms demonstrating a response such as death.

D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in Whole Effluent Toxicity tests is specified in the test method except the reference standard. All reference standards shall be prepared from chemicals which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in Appendix D.1 above.

D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit.

D.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid loss of cultures due to cross-contamination.

b) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s))and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory.

c) Instruments used for routine measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, and weight shall be calibrated, and/or standardized per manufacturer's instructions and Section D.1. Temperature shall be calibrated per section 5.9.4.2.1 All measurements and calibrations shall be documented.

d) Test temperature shall be maintained as specified in the methods manuals. The average daily temperature of the test solutions must be maintained within 1°C of the selected test temperature, for the duration of the test. The minimum frequency of measurement shall be

once per 24 hour period. The test temperature for continuous flow toxicity tests shall be recorded and monitored continuously.

e) Water used for culturing and testing shall be analyzed for toxic metals and organics annually or whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. The method specified analytes and concentration levels shall be followed.

f) New batches of food used for culturing and testing shall be analyzed for toxic organics and metals. If food combinations or recipes are used, analyses shall be performed on the final product upon the use of new lot of any ingredient. If the concentration of total organic chlorine exceeds 0.15 μg/g wet weight, or the total concentration of organochlorine pesticides plus PCBs exceeds 0.30 μg/g wet weight, or toxic metals exceeds 20 μg/g wet weight, the food must not be used.

g) Test chamber size and test solution volume shall be as specified in the methods manuals.

h) Test organisms shall be fed the quantity and type food specified in the methods manuals. They shall also be fed at the intervals specified in the test methods.

i) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test methods and shall be documented at least quarterly. For algal tests, the light intensity shall be measured and recorded at the start of each test.

j) At a minimum, during chronic testing DO and pH shall be measured daily in at least one replicate of each concentration. DO may be measured in new solutions prior to organism transfer, in old solutions after organisms transfer, or both.

k) All cultures used for testing shall be maintained as specified in the methods manuals.

l) Age and the age range of the test organisms must be as specified in the manuals.

m) The maximum holding time (lapsed time from sample collection to first use in a test) shall not exceed 36 hours without the permission of the permitting authority.

n) All samples shall be chilled to 4°C during or immediately after collection. They shall be maintained at a temperature range from just above the freezing temperature of water to 6°C and the arrival temperature shall be no greater than 6°C. Samples that are hand delivered to the laboratory immediately after collection (i.e., within 1 hour) may not meet the laboratory temperature acceptance criteria. In these cases, the laboratory may accept the samples if there is evidence (such as arrival on ice) that the chilling process has begun.

o) Organisms obtained from an outside source must be from the same batch.

D.3 MICROBIOLOGY TESTING

These standards apply to laboratories undertaking the examination of materials, products and substances involving microbiological analysis, recovery or testing. The procedures involve the culture media, the test sample and the microbial species being isolated, tested or enumerated.

a) Microbiological testing refers to and includes the detection, isolation, enumeration and identification of microorganisms and their metabolites, as well as sterility testing. It includes assays using microorganisms as part of a detection system and their use for ecological testing.

b) These standards are concerned with the quality of test results and not specifically with health and safety measures. In the performance of microbiological testing, laboratories must be aware of and have SOPs that conform with local, State, and national regulatory policies for the safety and health of personnel.

D.3.1 Positive and Negative Controls

a) Negative Controls

The laboratory shall demonstrate that the cultured samples have not been contaminated through sample handling/preparation or environmental exposure. These controls shall include sterility checks of media, blanks such as filtration blanks, bottle, and buffer blanks.

1) All blanks and uninoculated controls specified by the test method shall be prepared and analyzed at the frequency stated in the method.

2) A minimum of one uninoculated control shall be prepared and analyzed unless the same equipment set is used to prepare multiple samples. In such cases, the laboratory shall prepare a series of blanks using the equipment. At least one beginning and ending control shall be prepared, with additional controls inserted after every 10 samples.

3) Analyze a known negative culture.

b) Positive Controls

Positive controls demonstrate that the medium can support the growth of the test organism, and that the medium produces the specified or expected reaction to the test organism.

1) On a monthly basis each lot of media shall be tested with at least one pure culture of a known positive reaction and shall be included with the sample test batch.

2) If routine culturing is not part of a laboratory's testing and pre-prepared media are routinely used, strict control of the storage conditions and expiration date of media shall be maintained. A positive growth control from a known positive sample shall be run with each lot to ensure that the media support growth.

3) If the laboratory has at least one known positive result of the appropriate organism during the month, a separate positive control is not required.

D.3.2 Test Variability/Reproducibility

a) Duplicates - At least 5% of the suspected positive samples shall be duplicated. In laboratories with more than one analyst, each shall make parallel analyses on at least one positive sample per month.

b) Where possible, participation in, or organization of collaborative trials, proficiency testing, or interlaboratory comparisons, either formal or informal, must be done.

D.3.3 Method Evaluation

a) In order to demonstrate the suitability of a test method for its intended purpose, the laboratory shall demonstrate and document its ability to meet acceptance criteria either specified by the method or by the EPA or State program requirements. Acceptance criteria must meet or exceed these requirements and must demonstrate that the test method provides correct/expected results with respect to specified detection capabilities, selectivity, and reproducibility.

   1) Accepted (official) test methods or commercialized test kits for official test methods, or test methods from recognized national or international standard organizations, may not require a specific validation. Laboratories are required, however, to demonstrate proficiency with the test method prior to first use. This can be achieved by simultaneous, side-by-side analysis by several analysts.

   2) Qualitative microbiological test methods in which the response is expressed in terms of presence/absence, shall be validated by estimating, if possible, the specificity, and reproducibility. The differences due to the matrices must be taken into account when testing different sample types.

   3) The validation of microbiological test methods shall be performed under the same conditions as those for routine sample analysis. This can be achieved by using a combination of naturally contaminated products and spiked products with results that can be statistically analyzed to demonstrate that the test meets its intended purpose.

   4) All validation data shall be recorded and stored at least as long as the test method is in force, or if withdrawn from active use, for at least 5 years past the date of last use.

b) Laboratories shall participate in the Proficiency Test programs (interlaboratory) identified by NELAP (5.4.2.j or 5.5.3.4).

D.3.4 Test Performance

All growth and recovery media must be checked to assure that the target organisms respond in an acceptable and predictable manner (see D.3.1.b).

D.3.5 Data Reduction

a) The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

b) If the test method specifies colony counts, such as membrane filter or colony counting, then the ability of individual analysts to count colonies shall be verified at least once per month, by having two or more analysts count colonies from the same plate.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared in the laboratory from the different chemical ingredients, from commercial dehydrated powders or may be purchased ready to use.

b) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to 5.10.5. The laboratory shall retain all manufacturer-supplied "quality specification statements" which may contain such information as shelf life of the product, storage conditions, sampling regimen/rate, sterility check including acceptability criteria, performance checks including the organism used, their culture collection reference and acceptability criteria, date of issue of specification, or statements assuring that the relevant product batch meets the product specifications.

c) Distilled water, deionized water or reverse osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media solutions and buffers. The quality of the water shall be monitored for attributes such as pH, chlorine residual, specific conductance, metals, or heterotrophic plate count at the specified frequency and evaluated according to the stated standards. Records shall be maintained on all activities.

d) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's instructions or the test method.

e) All laboratory media shall be checked to ensure they support the growth of specific microbial cultures. In addition, selective media shall be checked to ensure they suppress the growth of non-target organisms. Media purchased pre-prepared from the manufacturer shall be checked monthly except when the use and maintenance of pure cultures is not part of laboratory procedures. In preference to using the commonly used streak method, it is better to use a quantitative procedure, where a known (often low) number of relevant organisms are inoculated into the medium under test and the recovery evaluated.

f) Each lot of detergent for laboratory use shall be checked to ensure that residues from the detergent do not inhibit or promote growth of microorganisms, for example with an inhibitory residue test.

D.3.7 Selectivity

a) All confirmation/verification tests specified by the test method shall be performed according to method protocols.

b) In order to demonstrate traceability and selectivity, laboratories shall use reference cultures of microorganisms obtained from a recognized national collection or an organization recognized by the assessor body.

1) Reference cultures may be subcultured once to provide reference stocks. Appropriate purity and biochemical checks shall be made and documented. The reference stocks shall be preserved by a technique which maintains the desired characteristics of the strains. Examples of such methods are freeze-drying, liquid nitrogen storage and deep-freezing methods. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be re-frozen and re-used.

2) Working stocks shall not be sequentially cultured more than five times except when:

i. it is required by standard test methods, or

ii. laboratories can provide documentary evidence demonstrating that there has been no loss of viability, no changes in biochemical activity and/or no change in morphology.

3) Working stocks shall not be subcultured to replace reference stocks.

4) A scheme for handling reference cultures is included in Figure D.1.

D.3.8 Constant and Consistent Test Conditions

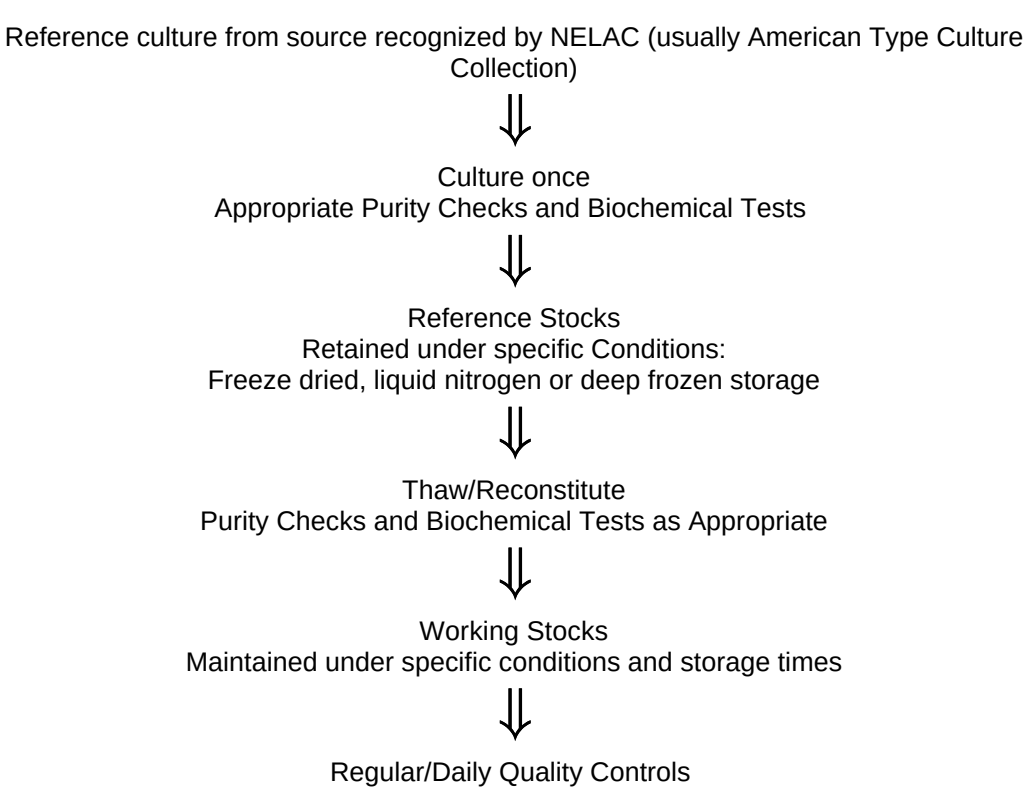
a) The laboratory shall devise an appropriate environmental monitoring program to indicate trends in levels of contamination appropriate to the type of testing being carried out. Acceptable background counts shall be determined and there shall be documented procedures to deal with situations in which these limits are exceeded.

b) Walls, floors, ceilings and work surfaces shall be non-absorbent and easy to clean and disinfect. Wooden surfaces of fixtures and fittings shall be adequately sealed. Measures shall be taken to avoid accumulation of dust by the provision of sufficient storage space by having minimal paperwork in the laboratory and by prohibiting plants and personal possessions from the laboratory work area.

c) Temperature measurement devices

1) Where the accuracy of temperature measurement has a direct effect on the result of the analysis, temperature measuring devices such as liquid-in-glass thermometers, thermocouple, platinum resistance thermometers used in incubators, autoclaves and other equipment shall be the appropriate quality to achieve the specification in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see 5.9.2). Calibration shall be done at least annually.

Figure D-1. USE OF REFERENCE CULTURES (BACTERIA)



2) The stability of temperature, uniformity of temperature distribution and time required to achieve equilibrium conditions in incubators, waterbaths, ovens and temperature controlled rooms shall be established, for example, position, space between and height of stacks of Petri dishes.

d) Autoclaves

1) The performance of each autoclave shall be initially evaluated by establishing its functional properties, for example heat distribution characteristics with respect to typical uses. Autoclaves shall be capable of meeting specified temperature tolerances. Pressure cookers fitted only with a pressure gauge are not recommended for sterilization of media or decontamination of wastes.

2) Records of autoclave operations including temperature and time shall be maintained. This shall be done for every cycle. Acceptance/rejection criteria shall be established and used to evaluate the autoclave efficiency and effectiveness.

e) Volumetric equipment such as automatic dispensers, dispenser/diluters, mechanical hand pipettes and disposal pipettes may all be used in the microbiology laboratory. Regular checks as outlined in Section 5.9.4.2.1 shall be performed and documented.

f) UV Sterilizers

1) Are to be tested quarterly for effectiveness with positives (either reference cultures or positive monitoring samples) and this is to include testing of the power output of the UV bulb.

g) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see Appendix D.1). Mechanical timers shall be checked regularly against electronic timing devices to ensure accuracy.

D.4 RADIOCHEMICAL TESTING

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive decay of analyte (or indicative daughters) and tracer isotopes where used. For the purpose of these standards procedures for the determination of radioactive isotopes by mass spectrometry (e.g. ICP-MS or TIMS) or optical (e.g. KPA) techniques are not addressed herein.

D.4.1 Negative Controls

a) Method Blank - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The method blank result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified method blank acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.a)19 and 20] will be followed. The occurrence of a failed

method blank acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

b) In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry the method blank shall be of similar counting geometry that is empty or filled to similar volume with ASTM Type II water to partially simulate gamma attenuation due to a sample matrix.

c) There shall be no subtraction of the required method blank [see D.4.1.a)] result from the sample results in the associated preparation or analytical batch. This does not preclude the application of any correction factor (e.g. instrument background, analyte presence in tracer, reagent impurities, peak overlap, calibration blank, etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

d) The method blank acceptance criteria [see 5.10.1.2.b)18] shall address the presumed aliquot size on which the method blank result is calculated and the manner in which the method blank result is compared to sample results of differing aliquot size.

D.4.2 Positive Controls

a) Laboratory Control Samples - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The laboratory control sample result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified laboratory control sample acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.a)19 and 20] will be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

b) Matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods which do not utilize an internal standard or carrier and for which there is a physical or chemical separation process and where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The matrix spike result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified matrix spike acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.a)19 and 20] will be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11]. The lack of sufficient sample aliquot size to perform a replicate analysis should be noted in the laboratory report.

c) The activity of the laboratory control sample and matrix spike analyte(s) shall be greater than ten times and less than one hundred times the *a priori* detection limit.

d) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration.

e) Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g. isotopic uranium: U-234, -235, and -238), only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified activity level each shall be assessed against the specified acceptance criteria.

f) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope, the laboratory control sample and matrix spike shall contain isotopes that represent the low (e.g. americium-241), medium (e.g. cesium-137) and high (e.g. cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples, the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

D.4.3 Test Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified replicate acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.a)19 and 20] will be followed. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

D.4.4 Other Quality Control Measures

a) Tracer - For those methods that utilize a tracer (i.e. internal standard) each sample result will have an associated tracer recovery calculated and reported. The tracer recovery for each sample results shall be one of the quality control measures to be used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified tracer recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.a)19 and 20] will be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

b) Carrier - For those methods that utilize a carrier (i.e., internal standard) each sample will have an associated carrier recovery calculated and reported. The carrier recovery for each sample shall be one of the quality control measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified carrier recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.a)19 and 20] will be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

D.4.5 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Capability - (section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel or method.

b) Proficiency Test Samples - The results of such analysis (5.4.2.j or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data. The providers of such proficiency test samples should conform to the requirements of ANSI N42.22.

D.4.6 Radiation Measurement System Calibration

Due to the stability and response nature of modern radiation measurement instrumentation, it is not typically necessary to calibrate these systems in the day of use manner done so for some types of chemical measurement instrumentation. As well due to the nature of some radiation measurement instrumentation calibrations, it may not be practical to calibrate in a day of use manner. In addition the calibration of modern radiation measurement instrumentation has significant differences from chemical measurement instrumentation. This section will address those practices that are necessary for proper calibration and those requirements of section 5.9.4.2 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) Calibration Curves

For those radiochemical methods that may require multiple standards for initial calibration (e.g. gas-proportional counting and liquid scintillation counting), the required number shall be addressed in the laboratory method manual [see 5.10.1.2.13] if not addressed in the method.

b) Calibration Curve Regression

Where linear regression is used to fit standard response or calibration standard results to a calibration curve the correlation coefficient shall be determined. Where non-linear regression is used to fit standard response or calibration standard results to a calibration curve the correlation coefficient should be determined.

c) Calibration Range

The requirements of 5.9.4.2.1.f) are not applicable to the performance of radiochemical methods given the non-correlated event nature of decay counting instrumentation.

d) Calibration Verification

The Laboratory Control Sample may fill the requirements for the performance of an initial calibration and continuing calibration verification standard as specified in sections 5.9.4.2.1 and 5.9.4.2.2. The calibration verification acceptance criteria shall be the same as specified for the Laboratory Control Sample.

e) Background Calibration- Background calibration measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives. These values are subtracted from the total measured activity in the determination of the sample activity.

1) For gamma spectroscopy systems, background calibration measurements shall be performed on at least a monthly basis.

2) For alpha spectroscopy systems, background calibration measurements shall be performed on at least a monthly basis.

3) For gas-proportional and scintillation counters, background calibration measurements shall be performed on a day of use basis.

f) Calibration - Instrument calibration shall be performed with reference standards as defined in section D.4.9.a. The standards shall have the same general characteristics (i.e. geometry, homogeneity, density, etc.) as the associated samples.

g) The frequency of calibration shall be addressed in the laboratory method manual [see 5.10.1.2.13] if not addressed in the method. A specific frequency (e.g. monthly) or observations from the associated control or tolerance chart, as the basis for calibration shall be specified.

D.4.7 Detection Limits

Note: To be addressed in the next Chapter Five revision.

D.4.8 Data Reduction

a) Refer to Section 5.10.6," Computers and Electronic Data Related Requirements," of this document.

b) Method Uncertainties - the laboratory shall have the ability to trace all sources of method uncertainties and their propagation to reported results. The ISO "Guide to the Expression of Uncertainty in Measurement" and/or the NIST Technical Note 1297 on "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results" should be used in this regard.

D.4.9 Quality of Standards and Reagents

a) The quality control program shall establish and maintain provisions for radionuclide standards.

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards should conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.

3) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the

certified value. The laboratory shall not use a value other than the decay corrected certified value.

b) All reagents used shall be analytical reagent grade or better.

D.4.10 Constant and Consistent Test Conditions

a) To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

b) Instrument performance checks - Instrument performance checks using appropriate check sources shall be performed on a regular basis and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the calibration has not changed. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the performance checks of the instrument. The check sources must provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel. For alpha and gamma spectroscopy systems, the instrument performance checks shall include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

1) For gamma spectroscopy systems, the performance checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

2) For alpha spectroscopy systems, the performance check for energy calibration shall be performed on a day of use basis and the performance check for counting efficiency shall be performed on at least a monthly basis.

3) For gas-proportional and scintillation counters, the performance checks for counting efficiency shall be performed on a day of use basis.

D.5 AIR TESTING

Analyses for Air Toxics shall follow the essential quality controls for chemistry outlined in Appendix D.1. For air testing, the blank, laboratory control sample and a desorption efficiency (such as charcoal tubes) shall be used. Matrix spikes and duplicate samples shall be used when feasible.

QUALITY SYSTEMS
APPENDIX E

ADDITIONAL SOURCES OF INFORMATION AND ASSISTANCE

-Non-Mandatory Appendix-

Appendix E - ADDITIONAL SOURCES OF INFORMATION
-Non-Mandatory Appendix-

Additional sources of information are available to assist laboratories in the design and implementation of a quality system. These materials may be found on the NELAC web page at www.epa.gov/ttn/nelac under the topic “Related Information”.